





Europäisches Patentamt
European Patent Office
Office européen des brevets



11 Publication number:

0 666 095 A1

12

EUROPEAN PATENT APPLICATION

21 Application number: 94101755.0

(5) Int. Cl.⁸: **S01D 39/16**, B01J 20/28, D04H 1/54

2 Date of filing: 05.02.94

Date of publication of application: 09.08.95 Bulletin 95/32

Designated Contracting States:
 AT BE ES FR GB IT NL SE

(7) Applicant: HOECHST CELANESE CORPORATION Route 202-206 North Somerville, N.J. 08876 (US)

Inventor: Groeger, Herbert Gunter 7716 Bradenton Drive Charlotte NC, 28210 (US)

Representative: von Kreisier, Alek, Dipl.-Chem. et al Patentanwälte von Kreisier-Selting-Werner Bahnhofsvorplatz 1 (Deichmannhaus) D-50667 Köln (DE)

(S) Particulate filter structure.

A particulate filter structure and method for making the structure are disclosed. The structure comprises a stable framework of relatively large denier fibers (12) having a lower melting component and a higher melting component. The larger denier fibers having a denier of from about 30 dpf or more and are bonded by the lower melting component at the crossover points (14). Smaller denier fibers (18), which preferably are of the same composition as the larger denier fibers (12), are immobilized onto the framework formed by the larger denier fibers (12) by applying heat. A particulate matter (16) such as activated carbon is immobilized primarily onto the smaller denier fibers (18), also by application of heat

Field of the Invention

This invention relates to dimensionally stable filter structures formed from thermoplastic fibers, and in particular to such filter structures having functional particulate matter incorporated therein.

Background of the Invention

Filter structures formed from thermoplastic fibers and having a functional particulate matter incorporated therein may be used for a variety of purposes. Such filter structures containing activated carbon have been used to adsorb noxious or harmful gases. Other activated particulates may be used depending on the function of the filter. For example, a biocide may be incorporated into a thermoplastic filter structure for the destruction of microbes passing through the filter. The variety of particulate matter and the functions associated therewith are extensively documented and are too extensive to list individually herein.

Examples of activated carbon filters for gas masks are disclosed in U.S. Patent Nos. 4,981,501 and 4,992,084. These patents disclose a three dimensional carrier framework for activated carbon particles having a diameter from 0.1 to 1 mm. The carrier framework is said to be composed of wires, monofilaments or stays, the distance between the components being at least twice as great as the diameter of the adsorbent particles. A large pore reticulated polyurethane foam is specifically disclosed as a carrier framework for granular adsorbent particles. These patents also disclose that the adsorbent particles can be affixed to heterofilic fibers having two coaxially arranged components wherein one component has a lower melting point than the other. However, no method for preparing a dimensionally stable carrier framework from such heterofilic fibers is disclosed.

It would be desirable to provide a dimensionally stable filter structure from thermoplastic fibers and an active particulate matter in which the carrier framework for the structure also carries a fibrous filter material in addition to the particulate filter material. Such a filter structure could provide greater surface area for attachment of particulate matter.

Summary of the Invention

This invention relates to a particulate filter structure having a high degree of dimensional stability and including a stable framework of relatively larger denier composite fibers, thermoplastic fibers of relatively smaller denier dispersed throughout and bonded to the framework, and an active particulate matter bonded to at least the smaller denier thermoplastic fibers. The larger denier fibers main-

tain the stability and permeability of the filter structure, which filter structure substantially is formed of the smaller denier fibers and the particulate matter. The larger denier fibers should have a denier of at least about 30 dpf and should comprise a relatively higher melting component and a relatively lower melting component. The lower melting component bonds the fibers of the framework at the cross-over points. The smaller denier fibers should have a denier of less than about 30 dpf and are dispersed throughout and bonded to the framework to immobilize the thermoplastic fibers and to provide a surface for attachment of active particulate matter. The active particulate matter is bonded to at least the smaller denier thermoplastic fibers.

In a more specific embodiment, the small and large denier fibers are formed of the same material and are sheath/core heterofilament fibers having a nylon sheath and a polyester core. A filter structure formed from such larger and smaller denier heterofilaments can be made having a thickness of from about 1.0 mm to 250 mm. The particulate matter can have a nominal particle diameter from 0.1 micron to 5 mm depending on the selection of the framework composite fibers and the smaller denier thermoplastic fibers.

The method for preparing such a particulate filter structure comprises preparing a framework of the relatively larger denier composite fibers having a denier of 30 dpf or greater, and thermally bonding these fibers at the cross-over points. Smaller denier thermoplastic fibers are dispersed into the framework and these fibers have a denier of less than about 30. The smaller denier fibers are immobilized onto the framework and a particulate matter is dispersed onto the framework and thermally bonded to at least the smaller denier fibers.

The smaller denier fibers can be dispersed into the framework by hydro-entanglement or air-entanglement or some other suitable method. Alternatively, a web of relatively small denier thermoplastic fibers can be formed and larger denier fibers can be integrated into the smaller denier web to provide dimensional stability.

In another specific method, a slurry of large and small denier fibers is placed into a mold and the solvent is removed to form a filter structure. Activated particulate matter may be included in the slurry or may be added after the filter structure is formed.

Brief Description of the Drawings

Figure 1 is a representation of a cross-section through a filter structure of the present invention.

Figure 2 is a representation of a cross-section through a representative thermoplastic fiber of the present invention.

45

25

30





Figure 3 is a representation of a portion of the filter structure of Figure 1 showing bonding of the fibers in the filter structure and bonding the particulate matter to the fibers of the filter structure.

Detailed Description of the Preferred Embodiments

Figure 1 illustrates broadly at 10 a representation of a cross-section through a portion of the filter structure of the invention claimed herein. The filter structure includes a stable framework of relatively larger denier composite structural fibers 12 that are thermally bonded at the cross-over points 14. By the term "structural fibers" is meant fibers of relatively larger denier that may be used to support a filter or fabric structure. These structural fibers have a denier of at least about 30 dpf.

The structural fibers may range in denier from about 30 dpf to 10,000 dpf or larger depending on the stiffness desired for providing a framework that is resistant to compression and is otherwise dimensionally stable and the size of the functional particulate 16 that is chosen. The composite fibers should be present in an amount sufficient to provide a structure to which may be bonded the relatively smaller denier fibers 18.

The composite fibers 12 have a higher melting component and a lower melting component. The higher melting component has a melting point at least about 20 °C higher than the relatively lower melting component. When heated above the melting point of the lower melting component, but below the melting point of the higher melting component, the lower melting component bonds the fibers of the framework together without affecting the structural integrity of the framework that is provided by the higher melting component.

Composite fibers suitable for use as structural fibers in the practice of the invention include bicomponent fibers in which the higher and lower melting components are arranged in a side-by-side relationship, or heterofilament fibers having either a concentric or eccentric sheath/core arrangement with the high melting component forming the core and the lower melting component forming the sheath. The term "heterofilaments" as used herein refers to both staple fiber and to continuous filament, unless otherwise specified. The term "fiber" as used herein also refers to both cut staple and to continuous filament, unless otherwise specified. In side-by-side fibers the two components, one higher melting and one lower melting, are simultaneously extruded through single orifice to form a fiber having two halves. In the concentric sheath/core arrangement, a higher melting component forms a core centered axially within a lower melting sheath. In the eccentric sheath/core arrangement, the higher melting component is not centered axially of the

fiber. Composite fibers are also sometimes referred to as bicomponent fibers.

Composite fibers having a lower melting nylon component and a higher melting polyester component have been found useful in the practice of the invention. For example, a sheath/core heterofilament (Figure 2) having a nylon sheath with a melting point from about 175-185 °C and a polyester core with a melting point of from about 240-256 °C has proven to be useful in the practice of the present invention.

Figure 2 is a representation of a cross-section through a heterofilament 20. Heterofilament fiber 20 is representative of the many types of composite manufactured fibers that may be used for the framework or for attachment of the active particulate of the filter structure shown at 10 in Figure 1

Fiber 20 is illustrated as a concentric sheath-core heterofilament fiber in which the sheath and the core each comprise about 50% of the cross-sectional area of the fiber. A range of area of the fiber occupied by the sheath is contemplated to be from about 20 to about 80%. The fiber has a lower melting nylon sheath 22 and a higher melting polyester core 24. The sheath should have a melting point that is at least about 20°C below the melting point of the core and should occupy about half the cross-section of the fiber to provide strong thermal bonding of the fiber structure without adversely affecting the integrity of the core. The core provides strength and integrity to the filter structure.

The framework of relatively large denier structural fibers 12 of the filter structure of the invention all include composite manufactured fibers as described above, having a lower melting component for thermal bonding of the structure at cross over points 14 and for immobilizing relatively smaller denier fibers 18. The skilled artisan should recognize that there are a wide variety of composite fibers having a higher melting component and a lower melting component that are suitable for the practice of the invention and that a heterofilament fiber having a nylon sheath and a polyester core is but one of the broad array of fibers available. The composite fibers 12 may also be in a wide variety of forms including crimped and non-crimped cut staple fibers, short cut staple, continuous filaments or blends thereof.

Smaller denier thermoplastic fibers 18 of the filter structure 10 are immobilized on the framework of larger denier fibers and provide, in addition to the particulate matter, active filtration of liquids and gases. These smaller denier fibers also greatly increase the surface available for immobilizing the functional particulates. These smaller denier fibers can range in denier from about 1 to 30 dpf. Smaller denier staple fibers and short cut staple are par-

50



ticularly useful in the filter structure of the invention, although continuous filaments are also contemplated. Fibers 18 should be present in an amount sufficient to immobilize the particulate mat-

ter and to provide a desired filtration at an accept-

able pressure drop across the filter.

The smaller denier thermoplastic fibers can be immobilized in the framework of larger denier fibers through the application of heat. Care should be exercised to avoid fusing the smaller fibers into a mass that could adversely impact pressure drop across the filter or otherwise reduce filter efficiency. It has been found useful in the practice off the invention to use smaller denier fibers that have the same components as the composite structural fibers forming the framework.

For example, if sheath/core heterofilaments of nylon and polyester are used to form the framework, then it is useful for bonding the structure and for immobilizing the smaller diameter fibers and the particulate matter if the smaller denier fibers are also sheath/core heterofilaments of nylon and polyester having similar melting points. This similarity in melting points simplifies bonding of the filter framework, bonding of the smaller diameter fibers to the framework, and bonding of the particulate matter to the filter structure. Bonding and immobilization of the smaller denier fiber to the larger denier fiber, and fusing of the particulate matter to the larger and smaller denier fibers, is much the same as described herein above with respect to the bonding the larger denier composite fibers to form a framework.

Figure 3 is a greatly enlarged representation of particulate matter 16 immobilized within a framework of larger denier and smaller denier fibers 12 and 18, respectively. The smaller denier fibers are immobilized on the framework of larger denier fibers at numerous points 26. The particles are fused primarily to the smaller denier fibers at numerous points 28, and may also be bonded to the larger denier fibers as shown at 30. As can be seen from Figure 3, the filter structure is a cage-like structure in which particulate matter may be entrapped and substantially precluded from migrating. Fusing of the particle to the individual fibers desirably is localized in that the fiber does not form a film over the particle or otherwise substantially reduce the surface area of the particle available for contact with a liquid or gas stream moving through the filter structure.

The particulate matter may be selected from a wide variety of substances having some function that is desirably incorporated into a fibrous structure. One of the most common is activated carbon. Other types of functional particulate matter includes silica, zeolite, molecular sieve, clay, alumina, ion exchange resin, organic metal catalyst, metal ox-

ide, biocide, fungicide, and virucide. For example, a fungicide particulate matter may be incorporated into a filter structure such as for an automobile climate control system to remove mildew and mildew odors from the circulated air. Biocides and virucides may be incorporated into filters for protection against microbial components. Particulate sizes may range from a small size of 0.1 micron for biocides and fungicides to 3 to 5 mm for other types of particulate matter, and vary in shape from irregular to circular cylinders up to about 50 mm in length. The foregoing list is intended to be representative of the wide variety of functional particulate matter available and not in limitation of the substances suitable for use in the practice of the present invention.

The filter structures of the present invention may be produced by a variety of processes. For example, a slurry of the larger denier fibers and smaller denier fibers may be formed in a solvent of water, acetone or other suitable hydrocarbon and placed into a mold. Particulate matter may or may not be included in the slurry, as desired. The liquid component of the slurry is removed, typically by vacuum, and through-air heat may be applied to dry the filter structure. If particulate matter was present in the slurry, then heat may also be applied to bond the filter structure and to bond particulate matter to the fibers. Otherwise, the particulate matter is then distributed into the dried structure. Activated carbon beads can be heated prior to application so that localized bonding takes place on contact with the fibers of the filter structure. The fibers of the filter structure are thereafter bonded. Alternatively, the particulate matter may be distributed cold and the entire structure and particulate bonded in one step.

For example, activated carbon beads may be fused to individual concentric sheath/core fibers of the filter structure of the invention by heating the particles above the melting point of the low melting sheath component of the fiber and then dispersing the particles into the filter structure. Activated carbon can also be applied cold and then heated for immobilization within the filter structure. Activated carbon particles and other inorganic oxides and hydrates have significantly lower specific heats than polymers and so heat quickly and result in localized fluidity of the polymer. A "spot weld" is produced with a thin, controlled layer of adhesive provided by the polymer that minimizes loss of surface area of the particle.

The filter structure of the invention can also be formed using dry forming methods such as carding or air laying of staple fibers or forming a web of continuous filaments. A web of the relatively larger denier fibers can be formed first and the smaller denier fibers and particulate matter dispersed

15

20

25

-

therein and immobilized. Alternatively, a web of the relatively smaller denier fibers can be formed and the larger denier fibers thereafter integrated into the web. Particulate matter can be applied and immobilized either before or after the larger denier fibers are integrated into the web to form the filter structure of the web. Also, the web can be formed from the relatively smaller denier fibers and the relatively larger denier fibers together, and particulate matter can be applied and immobilized either during or after the formation of the web.

The particulate matter may be applied to the web from scatter coaters, engraved rolls, or screen conveyors. An inclined ramp may be used to spread the particulate matter and to enmesh the particulate matter within the web. Suitable methods for distributing particulate matter into a web are described in applicant's copending United States patent application U.S.S.N 07/977995 filed November 18, 1992.

The invention has been described with reference to particular preferred embodiments as illustrated in the drawing. However, these embodiments should be-considered illustrative of and not in limitation of the invention claimed herein. On the contrary, the invention includes all alternatives, modifications, and equivalents that may be included within the scope and spirit of the invention as defined by the appended claims.

Claims

- A particulate filter structure characterized by a high degree of dimensional stability, said filter structure comprising:
 - a) a stable framework of relatively larger denier composite fibers thermally bonded at the cross over points to stabilize said framework, said composite fibers having a denier of at least about 30 dpf and comprising a relatively higher melting component and a relatively lower melting component, wherein said higher melting component has a melting point at least about 20 °C higher than said relatively lower melting component, and wherein said lower melting component bonds the fibers at the cross over points;
 - b) thermoplastic fibers of relatively smaller denier than said larger denier fibers dispersed throughout and bonded to the framework by the application of heat to immobilize said thermoplastic fibers and to provide a surface for attachment of active particulate matter, said smaller denier fibers having a denier of less than about 30 dpf;
 - c) active particulate matter bonded by the application of heat at least to said smaller

denier thermoplastic fibers to form said filter structure.

- The filter structure of Claim 1 wherein said larger denier composite fibers are staple fibers.
- 3. The filter structure of Claim 1 wherein said larger denier composite fibers are sheath/core heterofilament fibers comprising a nylon sheath and a polyester core.
- 4. The filter structure of Claim 3 wherein said nylon sheath has a melting point of from about 175 to 185 °C and said polyester core has a melting point of from about 240 to 256 °C.
- The filter structure of Claim 1 wherein said larger denier composite fibers are of from about 30 to 10,000 dpf.
- 6. The filter structure of Claim 1 wherein said larger denier composite fibers comprise about 10 percent or less of the total weight of the filter structure.
- The filter structure of Claim 1 wherein said smaller denier thermoplastic fibers are staple fibers.
- 8. The filter structure of Claim 1 wherein said smaller denier thermoplastic fibers are composite fibers having a relatively higher melting component, wherein said higher melting component has a melting point at least about 20°C higher than said relatively lower melting component, and wherein said lower melting component bonds the fibers to said support structure at the cross over points, said smaller denier fibers having a denier of from about 1 to 30 dpf.
- 9. The filter structure of Claim 8 wherein said smaller denier composite fibers are sheath/core heterofilament fibers comprising a nylon sheath and a polyester core.
 - 10. The filter structure of Claim 9 wherein said nylon sheath has a melting point of from about 175 to 185 °C and said polyester core has a melting point of from about 240 to 256 °C.
 - 11. The filter structure of Claim 1 having a thickness from about 1.0 mm to 250 mm.
 - 12. The filter structure of Claim 1 wherein said particulate matter is selected from among the group comprising activated carbon, silica,

5

50

15

20

25

30

45

50



zeolite, molecular sieve, clay, alumina, ion exchange resin, organic metal catalyst, metal oxide, biocide, fungicide, and virucide.

- 13. The filter structure of Claim 12 wherein said particulate matter has a nominal particle diameter of from about 0.1 micron to 5.0 mm.
- 14. A particulate filter structure characterized by a high degree of dimensional stability, said filter structure comprising:
 - a) a stable framework of sheath/core heterofilaments having a core with a melting point at least 20 °C higher than the sheath and having a denier of from about 30 to 10,000 dpf, said heterofilaments being thermally bonded at the cross over points by the application of heat to stabilize said framework;
 - b) sheath/core staple heterofilaments of relatively smaller denier having a denier of from about 1 dpf to 30 dpf dispersed throughout and bonded to said framework through the application of heat to immobilize said smaller denier heterofilaments and to provide a surface for attachment of active particulate matter; and
 - c) active particulate matter bonded by the application of heat to at least said smaller diameter heterofilaments to form said low resistance filter structure, whereby said larger denier heterofilaments comprise of about 10 percent or less of the total weight of said filter structure, said filter structure having a thickness of from about 1.0 mm to 250 mm.
- 15. The filter structure of Claim 14 wherein said relatively larger and said relatively smaller sheath/core heterofilaments are comprised of the same components.
- 16. The filter structure of Claim 15 wherein said sheaths comprise nylon having a melting point of from about 175 to 185 °C and wherein said cores comprise polyester having a melting point of from about 240 to 256 °C.
- 17. The filter structure of Claim 14 wherein said particulate matter is selected from among the group comprising activated carbon, silica, zeolite, molecular sieve, clay, alumina, ion exchange resin, organic metal catalyst, metal oxide, biocide, fungicide, and virucide.
- 18. An activated carbon filter structure characterized by a high degree of dimensional stability, said filter structure comprising:

- a) a stable framework of staple sheath/core heterofilaments having a polyester core with a melting point of from about 240 to 256 °C and a nylon sheath with a melting point of from about 175 to 185 °C and having a denier from about 30 dpf to 10,000 dpf, said heterofilaments being thermally bonded at the cross over points by the application of heat to stabilize said framework;
- b) sheath/core staple heterofilaments of relatively smaller denier having a polyester core with a melting point of from about 240 to 250 °C and a nylon sheath with a melting point of from about 175 to 185 °C, said smaller denier heterofilaments being bonded to said framework through the application of heat to immobilize said smaller denier heterofilaments and to provide a surface for attachment of active carbon; and
- c) active carbon particles bonded by the application of heat to at least said smaller denier heterofilaments to form said low resistance filter structure, whereby said larger denier heterofilaments comprise of about 10 percent or less of the total weight of said filter structure and said filter structure has a thickness from about 1.0 mm to 250 mm.
- 19. A method for preparing a particulate filter structure comprising:
 - a) preparing a framework of relatively larger denier composite fibers having a denier of 30 dpf or greater;
 - b) thermally bonding the fibers at the cross over points:
 - c) dispersing relatively smaller denier thermoplastic fibers into the framework, the smaller denier fibers having a denier of 30 or less;
 - d) immobilizing the smaller denier filaments within the framework;
 - e) dispersing particulate matter into the framework; and
 - f) thermally bonding the particulate matter at least to the smaller denier fibers.
- 20. The method of Claim 19 wherein the smaller denier fibers are dispersed into the framework by air entanglement.
- 21. The method of Claim 19 wherein the smaller denier composite fibers are dispersed into the framework by hydroentanglement.
- 22. The method of Claim 19 wherein the smaller denier fibers are composite fibers having a higher melting component and a lower melting component and are immobilized within the





framework by applying heat to bond the lower melting component to the larger denier fibers.

- 23. The method of Claim 22 wherein the particulate matter is immobilized by heating the particulate matter substantially above the melting point of the sheath but below the melting point of the core prior to dispersing the particulate matter into the framework so that the particulate bonds to the sheath.
- 24. A method for preparing a particulate filter structure comprising:
 - a) preparing a web of thermoplastic fibers having a denier of 30 or less;
 - b) integrating composite fibers having a denier of 30 or more into the web of thermoplastic fibers;
 - c) applying heat to thermally bond the larger denier composite fibers at the cross over points to form a stabilized framework and to bond the smaller denier thermoplastic fibers to the larger denier composite fibers, thereby immobilizing the smaller denier thermoplastic fibers within the framework;
 - d) applying particulate matter into the framework; and
 - e) immobilizing the particulate matter.
- 25. The method of Claim 24 wherein the composite fibers and the thermoplastic fibers are sheath/core heterofilament fibers having a lower melting sheath and a higher melting core and wherein the particulate matter is immobilized by heating the particulate matter substantially above the melting point of the sheath but below the melting point of the core prior to applying the particulate matter to the framework so that the sheaths bond to the particulate.

5

10

15

20

25

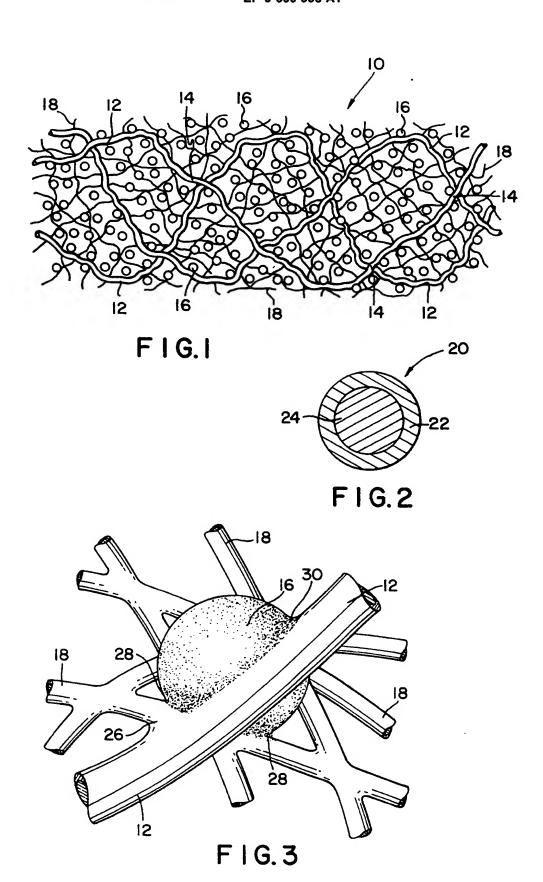
30

35

40

45

50





EUROPEAN SEARCH REPORT

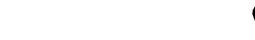
Application Number EP 94 10 1755

Category	Citation of document with of relevant p	indication, where appropriate, assages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.CL6)
X	EP-A-0 257 868 (MIN MANUFACTURING COMP/ * column 2. line 13		11-13,24	B01D39/16 B01J20/28 D04H1/54
A			5,8,14, 17,18	50 1112, 01
4		VON BLÜCHER ET AL.)	1-6, 11-14,17	
	* the whole documen			
A	EP-A-0 156 649 (MIN MANUFACTURING COMP/		1,2,5-7, 11-14, 17-20	
	* page 2, line 32 - * page 5, line 35 - * page 9, line 11 - * claims; figures *	- page 8, line 31 * - page 12, line 11 *		
A	FR-A-2 120 871 (TE	IJIN LIMITED)	1-4, 11-13	
	* page 2, line 13 - * page 7, line 6 - * page 11, line 28 * claims; figure 1	page 8, line 22 * - page 12, line 35 *		TECHNICAL FIELDS SEARCHED (Int.Cl.6) B01D B01J
A	WO-A-85 03013 (HUGHES AIRCRAFT COMPANY)		1,2,5-7, 11-13,17	DO4H
	<pre>* page 3, line 24 - * claims; figures *</pre>			
A	US-A-4 963 431 (AQUARIUM PHARMACEUTICALS, INC.)		1,2,5-7, 11-14, 17-19	
	* column 3, line 40 * figure 3 *) - column 5, line 39 *		
A	WO-A-93 01880 (LYD/ * claims *	ALL, INC.)	1-7,11	
		-/		
	The present search report has I	been drawn up for all claims	7	
	Place of search	Date of completion of the search		Examiner
	THE HAGUE	12 July 1994	Ste	vnsborg, N
X : par Y : par	CATEGORY OF CITED DOCUME ticularly relevant if taken alone ticularly relevant if combined with an ument of the same category	E : earlier patent of after the filing sother D : document cite	iple underlying the focument, but publicate in the application for other reasons	

EPO PORM 1503 03.82 (PO4C01)









EUROPEAN SEARCH REPORT

Application Number EP 94 10 1755

Category	Citation of document with in of relevant page	dication, where appropriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.CL6)
E	WO-A-94 11556 (HOEC CORPORATION) 26 May (filed 18 November * the whole document	1994 1992)	1-25	(indept)
				TECHNICAL FIELDS SEARCHED (Int.Cl.6)
1	he present search report has been	drawn up for all claims	1	
	lace of search	Date of completion of the search		Examiner
T	HE HAGUE	12 July 1994	Stevi	nsborg, N
: particu : particu docum	FEGORY OF CITED DOCUMENTS larly relevant if taken alone larly relevant if combined with another mother of the same category ogical background	E : earlier patent do	ole underlying the in- cument, but publish ate	vention

EPO FORM 1503 03.42 (POICOL)